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YTH TRANSLATION  
JP H3-295828

Translation of JP H3-295828

Application No.: H2-97344  
Application Date: April 12, 1990  
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**TITLE:** UP-CONVERSION GLASS

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**APPLICANTS:** HOYA Corp.

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**1. TITLE OF INVENTION**

Up-conversion glass

**3. PATENT CLAIMS**

**[Claim 1]**

Up-conversion glass which is formed by the oxide glass comprising heavy metal oxides and rare earth element oxides, wherein the maximum lattice vibration energy of the said oxide glass is characteristically at most  $1000\text{ cm}^{-1}$ .

**[Claim 2]**

The up-conversion glass described in Claim 1, which comprises 20 to 85 mol% of the heavy metal oxides and 0.1 to 30 mol% of the rare earth oxide.

**3. DETAILED EXPLANATION OF INVENTION**

**[Industrial Application Fields]**

The present invention relates to the up-conversion glass which is suitable for the display, the detector of IR ray and IR laser, and for the up-conversion laser material.

**[Conventional Techniques]**

The material which converts the irradiated IR beam into the visible light and emits the fluorescence or the laser beam (below, it is called an up-conversion material) has a high utilization value as the display, as the detector of IR ray and IR laser, and for the visible laser beam material, therefore, the various R&D works have been promoted.

For example, JP Kohyo 63-503495 discloses the crystal of which major component is potassium fluoride, doped with the active ion,  $\text{Er}^{3+}$ .

Further, "Electronics Letters, 23 (1), 32-34, January 1987" describes the visible luminescence obtained by the  $\text{Er}^{3+}$  up-conversion in the fluoride glass and  $\text{CaF}_2$  crystal doped with  $\text{Yb}^{3+}$  and  $\text{Er}^{3+}$ .

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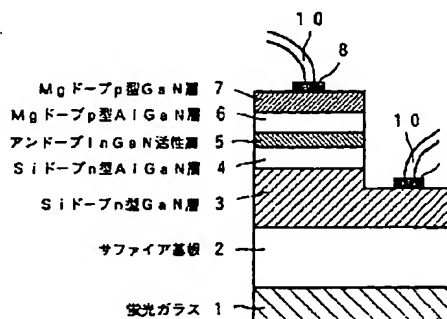
(54) LIGHT-EMITTING DIODE AND ITS  
MANUFACTURING METHOD

(57) Abstract:

PROBLEM TO BE SOLVED: To obtain a mixed color light-emitting diode that allows the material and process of resin mold that are exactly the same as those of the conventional monochromatic light-emitting diode to be applied, and prevents the decrease in emission efficiency and increase in a drive voltage as compared with the conventional monochromatic light-emitting diode.

SOLUTION: Semiconductor layers 3-7 are stacked on a substrate 2 having at least a layer of fluorescent glass layer 1, or at least a layer of fluorescent glass layer 11 is stacked or joined to a compound semiconductor wafer where semiconductor layers 13-17 are stacked and formed.

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Among these up-conversion materials, the up-conversion material having the glass as the medium (below, it is called an up-conversion glass) has the following advantages, compared to the up-conversion material having the  $\text{CaF}_2$  crystal as the medium: (1) an easy manufacturing, (2) a low cost, and (3) a possible fiber fabrication. Further, in the up-conversion glass, the absorption spectrum of the rare earth ion within the glass is broad, thus the variation in the absorption efficiency accompanied by the excitation light wavelength fluctuation is small. As a result, even when the semiconductor laser, of which output wavelength is easily influenced by the temperature and the current, is used as the excitation light, the relatively stable power can be obtained.

[Problems Solved by the Invention]

The conventional fluoride glass type up-conversion glass presents the IR-visible light conversion efficiency comparative to that of the up-conversion material having the  $\text{CaF}_2$  crystal as the medium, therefore, it is the practical material from this view point. However, the fluoride glass has the problems of a low chemical durability and mechanical strength, which makes it less useful for the practical application. Further, in the fluoride glass, the compositional region at which the stable glass is formed is extremely narrow, thus the compositional variation for the large alteration of the characteristics has been difficult. Furthermore, in the fluoride glass, the glass manufacturing atmosphere control and the forming were also difficult.

In the said Electronic Letters, the IR-visible light conversion efficiency values measured for the silicate glass and phosphate glass doped with  $\text{Yb}^{3+}$  and  $\text{Er}^{3+}$  are also described. The IR-visible light conversion efficiency values in these glasses are  $1/100,000^{\text{th}}$  to  $1/10,000^{\text{th}}$  of the fluoride glass type up-conversion glass, therefore, the up-conversion phenomenon is hardly occurring in these glasses.

Therefore, the purpose of the present invention is to offer the up-conversion glass which possesses the practical level of the IR-visible light conversion efficiency value, the superior chemical durability and mechanical strength, and the easy manufacturing and forming property.

[Method to Solve the Problems]

The present invention was made to achieve the said goal and the up-conversion glass of the present invention is characteristically formed by the oxide glass comprising the heavy metal oxides and the rare earth element oxides, wherein the maximum lattice vibration energy of the said oxide glass is at most  $1000 \text{ cm}^{-1}$ .

Below, the present invention is interpreted in details.

The up-conversion glass of the present invention is characteristically formed by the oxide glass which comprises the heavy metal oxides.

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Examples of the heavy metal oxide are: PbO, CdO, Bi<sub>2</sub>O<sub>3</sub>, Ga<sub>2</sub>O<sub>3</sub>, TeO<sub>2</sub>, Sb<sub>2</sub>O<sub>3</sub>, As<sub>2</sub>O<sub>3</sub>, and GeO<sub>2</sub>. The up-conversion glass of the present invention comprises at least one type of these heavy metal oxides. The desirable content of the heavy metal oxides is from 20 to 85 mol%. At the content of less than 20 mol%, it is difficult for the maximum lattice vibration energy to be at most 1000 cm<sup>-1</sup> at the practical level. The content over 85 mol% causes the difficult glass formation. The particularly desirable content of the heavy metal oxides is from 25 to 82 mol%.

Further, the up-conversion glass of the present invention also comprises the rare earth element oxides and the rare earth element which constructs the rare earth element oxide is the ion causing the up-conversion phenomenon (up-conversion active ion).

Examples of the rare earth element are: Ce, Pr, Nd, Pm, Sm, Eu, Tb, Dy, Ho, Er, Tm, Yb, and Lu. The rare earth element oxides may be the oxide of these rare earth elements. The desirable content of the rare earth element oxides is from 0.1 to 30 mol%. The content less than 0.1 mol% is not practical due to the low IR-visible conversion efficiency and the content over 30 mol% causes the difficult glass forming. The particularly desirable content of the rare earth element oxides is from 1 to 20 mol%.

Further, the up-conversion glass of the present invention may also comprise alkali metal oxides such as Li<sub>2</sub>O, Na<sub>2</sub>O, K<sub>2</sub>O, Rb<sub>2</sub>O, and Cs<sub>2</sub>O, alkali earth oxides such as MgO, CaO, SrO, and BaO, and other components which are usually utilized as the glass components such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, P<sub>2</sub>O<sub>5</sub>, ZnO, WO<sub>3</sub>, and MoO<sub>3</sub>, in addition to the said heavy metal oxides and the rare earth element oxides.

The maximum lattice vibration energy of the up-conversion glass of the present invention is limited to at most 1000 cm<sup>-1</sup>. The reason to limit the maximum lattice vibration energy to at most 1000 cm<sup>-1</sup> is interpreted below.

In the up-conversion glass formed by the oxide glass, the intensity of the luminescence generated by the IR-visible light conversion increases when the phonon energy which concerns to the multi-phonon relaxation is smaller. This phonon energy concerning to the multi-phonon relaxation is, for example in the up-conversion glass comprising Eu<sup>3+</sup>, is equivalent to the energy difference between the electron transfer peak (<sup>7</sup>F<sub>0</sub> → <sup>5</sup>D<sub>2</sub> transfer) which appears at the measurement of the glass excitation spectrum by monitoring the luminescence by the <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>2</sub> transfer (612 nm) and the phonon side band peak which appears at the higher energy side of this electron transfer peak. In addition, in the oxide glass type up-conversion glass comprising the rare earth elements other than Eu, the energy difference between the electron transfer peak and the phonon side band peak also corresponds to the phonon energy concerning to the multi-phonon relaxation.

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These electron transfer peak and phonon side band peak can be measured by using the ordinary fluorescence spectrophotometer. The glasses comprising the rare earth elements other than Eu have the rare earth ion electron transfer band at the position where the phonon side band peak appears. Therefore, the phonon side band is covered by the band originated from the electron transfer and the identification of the phonon side band is difficult. Further, the glass phonon energy is independent from the type of the doped rare earth elements. Therefore, the phonon energy of the glass comprising the rare earth elements other than Eu is same as the phonon energy of the said glass of which rare earth element is replaced by Eu. The production of the multiple glasses of which compositions are exactly the same except the rare earth elements is not always easy unless they are prepared all at the same time. Therefore, in the glass comprising the rare earth elements other than Eu, the accurate measurement of the phonon energy concerning to the multi-phonon relaxation is difficult.

The phonon energy value concerning to the multi-phonon relaxation can be also obtained by the lattice vibration energy which is obtained from the glass Raman scattering spectrum (Physical Review, B16, p10 (1977)). Also in this case, as similar to the phonon energy concerning to the multi-phonon relaxation, the intensity of the luminescence caused by the IR-visible light conversion increases when the maximum lattice vibration energy is smaller. And in the oxide up-conversion glass, the maximum lattice vibration energy exceeding  $1000\text{ cm}^{-1}$  results in either no exhibition of the up-conversion phenomenon or the exhibition of a very weak effect.

Therefore, the up-conversion glass of the present invention limits the maximum lattice vibration energy to be at most  $1000\text{ cm}^{-1}$ . When the maximum lattice vibration energy is at most  $1000\text{ cm}^{-1}$ , the rare earth ion excited by the IR has small energy loss caused by the non-radiative transition from the excited level and the up-conversion phenomenon is remarkably exhibited.

The maximum lattice vibration energy of the present specification which is obtained from the Raman scattering spectrum was measured by the following procedure. All six faces of the sample (the size of  $10 \times 25 \times 4\text{ mm}$ ) were polished and the laser beam of  $514.5\text{ nm}$  and  $60\text{ mW}$  output was irradiated on one surface of the polished sample with the incident angle of  $45^\circ$ . The scattered light was detected by the Raman spectroscopy to obtain the Raman scattering spectrum. The wave number of the scattering band peak at the highest wave number side corresponds to this energy.

The up-conversion glass of the present invention which comprises the heavy metal oxides and the rare earth element oxides as the essential components and has the maximum glass lattice vibration energy of at most  $1000\text{ cm}^{-1}$  may employ the oxides, carbonates, and nitrates of the heavy metals to be comprised as the starting raw materials of the heavy metal oxides, and the oxides, carbonates, and nitrates of the rare earth elements to be comprised as the starting raw materials of the rare earth element oxides. In addition, the ordinary glass raw materials such as the oxides,

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carbonates, and nitrates of the arbitrary components such as the alkali metal oxides and the alkali earth metal oxides may be employed as necessary. These starting raw materials are mixed so that the final glass composition will have the desired composition and the glass is obtained from this mixture by the ordinary melting method of the oxide glass.

Therefore, the up-conversion glass of the present invention does not have the problems of the atmosphere control and the forming difficulty which occur during the preparation of the fluoride glass type up-conversion glass and offers the up-conversion glass which has a superior chemical durability and mechanical strength to the fluoride glass type up-conversion glass and also the variously formed shapes. Further, the wide compositional region for the glass forming enables the alteration in the composition, which results in the up-conversion glass with various characteristics.

[Examples]

Below, the examples of the present invention are interpreted. However, the present invention is not limited to these examples.

Example 1

By using  $\text{TeO}_2$  as the starting raw material of the heavy metal oxide,  $\text{Er}_2\text{O}_3$  as the starting raw material of the rare earth element oxide, and  $\text{Na}_2\text{CO}_3$  as the starting raw material of the arbitrary component, the mixture of the starting raw materials was prepared so that the final glass composition is the composition shown in Table 1. The mixture was made molten at 900 °C for 60 minutes in the air to form the glass melt and the obtained glass melt was poured on an iron sheet to solidify. Then, the annealing at the rate of 30 °C/h resulted in the oxide type up-conversion glass.

From the obtained up-conversion glass, the sample (10 x 25 x 4 mm size) was cut and six faces of this sample were polished and the laser beam of 514.5 nm and 60 mW output was irradiated on one surface of the polished sample with the incident angle of 45°. The scattered light was detected by the Raman spectrometer, NR-1000 manufactured by Nippon Bunko Kogyo K. K. to obtain the Raman scattering spectrum. The maximum energy of the glass lattice vibration (below called as the maximum lattice vibration energy) was obtained by the method described earlier and obtained to be 680  $\text{cm}^{-1}$  as shown in Figure 1. Here in Figure 1, the peak near 1300  $\text{cm}^{-1}$  (the peak section is not shown in the figure) is assigned to the  $\text{Er}^{3+}$  fluorescence.

Further, the said six-face polished sample used for the measurement of the maximum lattice vibration energy was irradiated with the excitation light, 800 nm IR semiconductor laser, and the fluorescence spectrum was measured. The fluorescence spectrum shown in Figure 2 has the peak at 550 nm, which is the fluorescence caused by the IR-visible light conversion. At this time, the fluorescence spectrum was measured by the following procedure. As shown in Figure 3, the laser beam from the excitation light source (IR semiconductor laser) 1 is pulsed by the chopper 2. Then the

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pulsed light is entered into the sample 4 by the mirror 3 and the visible light (fluorescence) from the sample 4 was focused by the lens 5 and entered to the spectrophotometer 6 (R 320 monochrometer manufactured by Jobin-Yvon). The light from the spectrophotometer 6 was detected by the photomultiplier 7 (R 2228 manufactured by Hamamatsu Photonics) cooled by liquid nitrogen. The result was amplified by the lock-in amplifier 8 (5208 manufactured by EG&G) and recorded by the recorder 9. The lock-in amplifier 8 was also connected to the chopper 2 in order to adjust the phase of the signal light and the reference light.

Further, the obtained up-conversion glass was measured for the fluorescence intensity caused by the IR-visible light conversion at room temperature (below referred to as the IR-visible light conversion fluorescence intensity), the chemical durability, and the destruction tenacity by the following procedure. The measurement results are shown in Table 1.

- IR-visible light conversion fluorescence intensity  
The sample (10 x 25 x 4 mm size) was cut from the obtained up-conversion glass and the six faces of this sample were polished. Then by using the device shown in Figure 3, one face of the sample was irradiated by the 800 nm IR semiconductor laser as the excitation light and the fluorescence spectrum was measured.
- Chemical durability  
Based on the measurement method of the optical glass chemical durability (powder method) regulated by the Japan Optical Glass Industry Standard in JOGIS-1975, the water resistance Dw (unit in wt%) was measured.
- Destruction tenacity  
The sample (5 x 5 x 30 mm size) was cut from the obtained up-conversion glass and one rectangular face was polished. Then a certain size crack was generated on the polished surface and the anti-bending strength was obtained by the three-point bending test. The destruction tenacity strength [ $K_{Ic}$  (units in  $\text{MPam}^{1/2}$ )] was normalized by the crack shape.

Examples 2 through 7

By using  $\text{Ga}_2\text{O}_3$ ,  $\text{PbO}$ ,  $\text{Bi}_2\text{O}_3$ ,  $\text{GeO}_2$ , and  $\text{TeO}_2$  as the starting raw materials of the heavy metal oxides,  $\text{Er}_2\text{O}_3$  as the starting raw material of the rare earth element oxide, and  $\text{Na}_2\text{CO}_3$ ,  $\text{SrCO}_3$ ,  $\text{WO}_3$ , and  $\text{Li}_2\text{CO}_3$  as the starting raw materials of the arbitrary components as necessary, the mixtures of the starting raw materials were prepared so that the final glass compositions are the compositions shown in Table 1. The mixture was made molten at 1100 to 1200 °C for 30 to 60 minutes in the air to form the glass melt and the obtained glass melt was poured on an iron sheet to solidify. Then, the annealing at the rate of 30 °C/h resulted in the oxide type up-conversion glass.



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Each of the obtained up-conversion glasses was measured for the maximum lattice vibration energy, the IR-visible light conversion fluorescence intensity, the chemical durability, and the destruction tenacity by the same procedures described in Example 1. The measurement results are shown in Table 1.

Comparison Example 1

As the starting raw materials,  $\text{ZrF}_4$ ,  $\text{BaF}_2$ ,  $\text{LaF}_3$ ,  $\text{AlF}_3$ ,  $\text{NaF}$ , and  $\text{ErF}_3$  were employed and the mixture was prepared so that the final glass composition is the composition shown in Table 1. The mixture was made molten at 875 °C for 60 minutes in the argon atmosphere and the obtained glass melt was cooled within the crucible at the rate of 30 °C/h to obtain the fluoride glass type up-conversion glass.

The obtained up-conversion glass was measured for the maximum lattice vibration energy, the IR-visible light conversion fluorescence intensity, the chemical durability, and the destruction tenacity by the same procedures described in Examples 1 though 7. The measurement results are shown in Table 1.

Comparison Example 2

As the starting raw materials,  $\text{P}_2\text{O}_5$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{K}_2\text{CO}_3$ ,  $\text{MgCO}_3$ ,  $\text{BaCO}_3$ , and  $\text{Er}_2\text{O}_3$  were employed and the mixture was prepared so that then final glass composition is the composition shown in Table 1. The mixture was made molten at 1200 °C for 60 minutes in the air and the obtained glass melt was poured on an iron sheet and solidified. Then the annealing at the rate of 30 °C/h resulted in the phosphate glass.

The obtained phosphate glass was measured for the maximum lattice vibration energy, the IR-visible light conversion fluorescence intensity, the chemical durability, and the destruction tenacity by the same procedures described in Examples 1 though 7. The measurement results are shown in Table 1.

Table 1

	Up-conversion glass composition	Max. lattice vibration energy (cm <sup>-1</sup> )	IR-VIS fluorescence intensity	Dw (wt%)	K <sub>IC</sub> (MPam <sup>1/2</sup> )
Ex. 1	69 TeO <sub>2</sub> – 30 Na <sub>2</sub> O – 1 Er <sub>2</sub> O <sub>3</sub>	680	27 x 10 <sup>-5</sup>	0.07	0.6
Ex. 2	55 SrO – 2 Ga <sub>2</sub> O <sub>3</sub> – 20 Er <sub>2</sub> O <sub>3</sub>	600	32 x 10 <sup>-5</sup>	0.11	0.7
Ex. 3	62 PbO – 20 Bi <sub>2</sub> O <sub>3</sub> – 17.5 Ga <sub>2</sub> O <sub>3</sub> – 0.5 Er <sub>2</sub> O <sub>3</sub>	600	15 x 10 <sup>-5</sup>	0.05	0.6
Ex. 4	55 GeO <sub>2</sub> – 35 Na <sub>2</sub> O – 10 Er <sub>2</sub> O <sub>3</sub>	900	25 x 10 <sup>-5</sup>	0.03	0.7
Ex. 5	60 GeO <sub>2</sub> – 25 Na <sub>2</sub> O – 5 Er <sub>2</sub> O <sub>3</sub>	900	30 x 10 <sup>-5</sup>	0.02	0.6
Ex. 6	75 GeO <sub>2</sub> – 20 Na <sub>2</sub> O – 5 Er <sub>2</sub> O <sub>3</sub>	900	15 x 10 <sup>-5</sup>	0.04	0.7
Ex. 7	53 TeO <sub>2</sub> – 25 WO <sub>3</sub> – 16 Li <sub>2</sub> O – 4 PbO – 2 Er <sub>2</sub> O <sub>3</sub>	920	40 x 10 <sup>-5</sup>	0.04	0.5
C.Ex 1	53 ZrF <sub>4</sub> – 20 BaF <sub>2</sub> – 3 LaF <sub>3</sub> – 3 AlF <sub>3</sub> – 20 NaF – 1 ErF <sub>3</sub>	500	100 x 10 <sup>-5</sup>	0.24	0.5
C.Ex 2	60 P <sub>2</sub> O <sub>5</sub> – 7 Al <sub>2</sub> O <sub>3</sub> – 17 K <sub>2</sub> O – 2 MgO – 13 BaO – 1 Er <sub>2</sub> O <sub>3</sub>	1270	5 x 10 <sup>-8</sup>	0.08	0.3

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As apparent from Table 1, the maximum lattice vibration energy of the up-conversion glass in Examples 1 through 7 is at most  $1000\text{ cm}^{-1}$  and their IR-visible light conversion fluorescence intensity at room temperature is comparative to that of the fluoride glass type up-conversion glass in Comparison Example 1. Therefore, the practical IR-visible light conversion efficiency was confirmed.

Further, the Dw values of the up-conversion glasses in Examples 1 through 7 were from 0.02 to 0.11 wt%, which is much smaller than the Dw value (0.24 wt%) of the fluoride glass type up-conversion glass in Comparison Example 1. Thus the superior chemical durability was demonstrated.

Further, the up-conversion glasses in Examples 1 through 7 have the  $K_{IC}$  value ranging from 0.5 to 0.7  $\text{MPam}^{1/2}$ , which is larger than the  $K_{IC}$  value ( $0.5\text{ MPam}^{1/2}$ ) of the fluoride glass type up-conversion glass in Comparison Example 1. Thus the superior destruction tenacity was demonstrated. Further, this indicates the superior mechanical strength.

Phosphate glass of Comparison Example 2 is superior to the up-conversion glasses in Examples 1 through 7 and in Comparison Example 1 from the view points of the chemical durability and the destruction tenacity [**Note from the Translator-** if the larger  $K_{IC}$  value indicates the better strength as stated above, the destruction tenacity of the phosphate glass ( $0.3\text{ MPam}^{1/2}$ ) is not superior]. However, it has very little IR-visible light conversion fluorescence intensity,  $5 \times 10^{-8}$ , therefore it is not a practical up-conversion glass.

Further, the IR-visible light conversion fluorescence intensity in each of the up-conversion glasses from Examples 1 to 7 has little variation accompanied by the external environmental temperature variation. In other words, it has little temperature dependency and offers the IR-visible light conversion fluorescence with a stable luminescence intensity compared to the fluoride glass type up-conversion glass which has a high temperature dependency.

[Effects of Invention]

As interpreted above, the up-conversion glass of the present invention possesses the practical IR-visible light conversion efficiency, a superior chemical durability and mechanical strength, and the easy manufacturing and forming property. In addition, the up-conversion glass of the present invention has little temperature dependency in its IR-visible light conversion fluorescence intensity.

Therefore, according to the present invention, the up-conversion glass practically usable as the display, the detector of IR ray and IR laser, and for the up-conversion laser material at room temperature can be obtained easily and by selecting the characteristics appropriately.

4. SIMPLE EXPLANATION OF FIGURES

Figure 1 is the Raman scattering spectrum to obtain the lattice vibration energy of the up-conversion glass obtained in Example 1 of the present invention.

Figure 2 is the fluorescence spectrum of the up-conversion glass obtained in Example 1 of the present invention.

Figure 3 is the schematic diagram of the instrumentation used to obtain the fluorescence spectrum shown in Figure 2.

4 - sample (up-conversion glass)

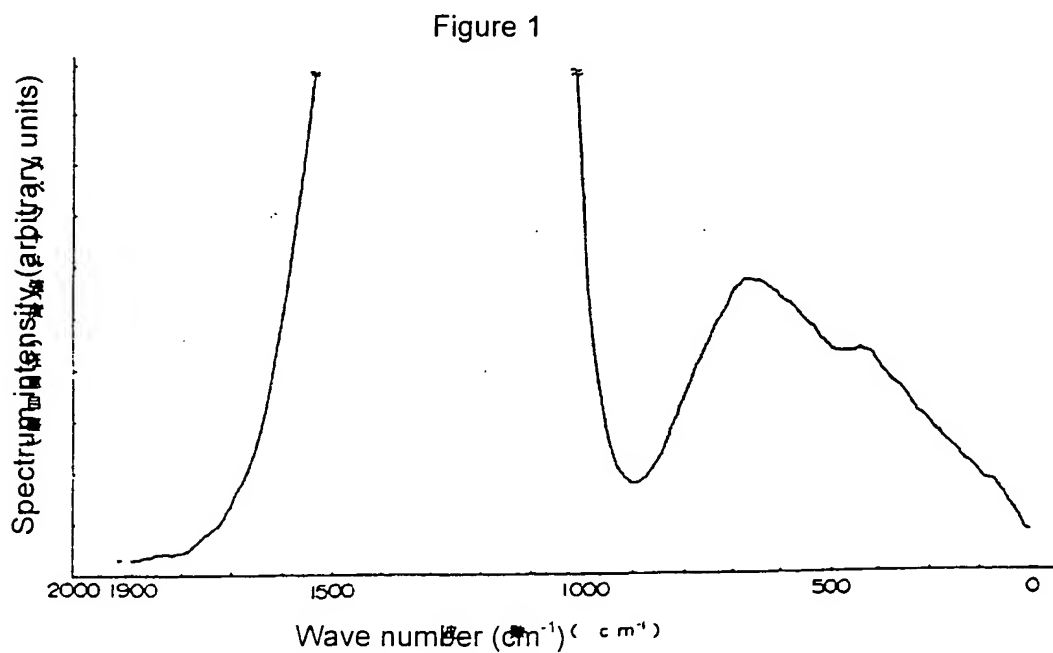


Figure 2

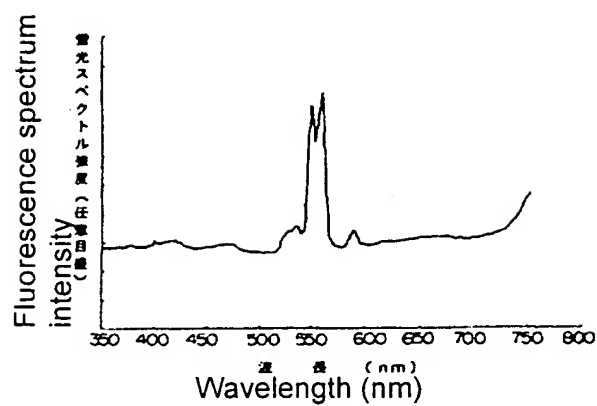


Figure 3

